

US EPA ARCHIVE DOCUMENT

**ANTIMONY OXIDE LISTING BACKGROUND
DOCUMENT FOR THE INORGANIC CHEMICAL
LISTING DETERMINATION**

This Document Contains No Confidential Business Information

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1. SECTOR OVERVIEW

1.1 SECTOR DEFINITION, FACILITY NAMES AND LOCATION

Antimony oxide currently is produced in the United States by four significant manufacturers using both direct and indirect methods¹. **Table 1.1** lists the name and location of the four manufacturers along with the production process employed by the facility.² **Figure 1.1** shows the geographic location of the facilities listed in Table 1.1.

Table 1.1 Antimony Oxide Producers

Facility	Location	Production Process
1. Great Lakes Chemical Corporation	7418 FM 1472 Laredo, TX 78045	Direct Process
		Indirect Process
2. Laurel Industries Inc.	780 S 16 th Street PO Box 1516 La Porte, TX 77571	Direct Process
		Indirect Process
3. Amspec Chemical Corporation	751 Water Street Gloucester City, NJ 08030	Direct Process
		Indirect Process
4. United States Antimony	1250 Prospect Creek Road Thompson Falls, MT 59873	Indirect Process

1.2 PRODUCTS, PRODUCT USAGE AND MARKETS

The chemical formula for antimony (III) oxide, also known as antimony trioxide, is Sb₂O₃. In pure form, it is a white powder and has a molecular weight of 291.52 g/mol. It has a melting point of 655 C and a boiling point of 1425 C.³

Antimony oxide appears as white, polymorphic crystals. It is slightly soluble in water and soluble in potassium hydroxide and dilute hydrochloric, nitric, and sulfuric acids.⁴

¹ Two additional manufacturers (Cerac, Inc. and Schumacher) were not evaluated because of their small production volume (906 gm (1997) and 0.36 MT respectively) and because they purify commercial grade product.

² Environmental Protection Agency, RCRA § 3007, Survey of the Inorganic Chemicals Industry.

³ <http://www.scorecard.org/chemical-profiles/html/antimony/html>.

⁴ Ibid.

Figure 1.1 Geographic Distribution of Antimony Oxide Producers¹



¹ See **Table 1.1** for facility name and location.

Antimony oxide typically is used as a flame retardant in plastics and textiles; a smoke suppressant; a stabilizer for plastics; a pigment constituent for coatings and plastics; an opacifier in glass, ceramics, and vitreous enamels; and as a coating for titanium dioxide pigments and chromate pigment.

1.3 PRODUCTION, PRODUCT AND PROCESS TRENDS

The major use for antimony oxide is as a flame retardant. Approximately 20,000 metric tons of antimony oxide were used in 1990 for this purpose. The production has approximately doubled since this industry was studied in 1985.⁵

In 1980, domestic production met consumer demand. At that time, the growth of the antimony oxide industry was expected to parallel the overall economy. The U.S. exported 1,353 tons of antimony oxide product in 1988.

The U.S. relies heavily on imports of antimony metal and ore in the production of antimony oxide. This is because domestic ore has high levels of arsenic, making use of this ore unattractive. In 1988, a total of 33,106 tons of ore was imported into the U.S., with China being the largest supplier. Some other major sources for this low arsenic ore are Central America and South Africa.⁶ Antimony ingots are also imported from China and Russia.

Based on 1980 information, only ore low in arsenic is processed because no practical and economical method for handling and disposing of arsenic-bearing residues has been developed. (It is unknown whether this is still the convention.) If an arsenic disposal method is developed, then less expensive domestic ore may be used.⁷

2. DESCRIPTION OF MANUFACTURING PROCESSES

2.1 PRODUCTION PROCESS DESCRIPTION

Antimony oxide can be prepared using one of two processes: the direct and the indirect. In this section, general descriptions of these two processes are provided. The process flow diagrams for these general production descriptions are presented in **Figure 2.1**.

Antimony Oxide Production via the Indirect Method

⁵ EPA, Multi-Media Assessment of the Inorganic Chemicals Industry, Versar, August 1980

⁶ Environmental Health Center: Environmental Writer, "Antimony Chemical Backgrounder", <http://safety.webfirst.com/ehc/EW.chems/antimony.htm>

⁷ EPA, Multi-Media Assessment of the Inorganic Chemicals Industry, Versar, August 1980

In this process, antimony metal (sources include ingots, ore, and sodium antimonate) is melted in a furnace. The molten metal is burned producing an antimony oxide vapor. The vapors are quenched with air and then cooled in hairpin coolers. Antimony oxide sublimes directly from a vapor to a white powdery solid. A filtering method, such as a baghouse, is used to recover the antimony powder. The antimony oxide powder is then blended and packaged for shipment.

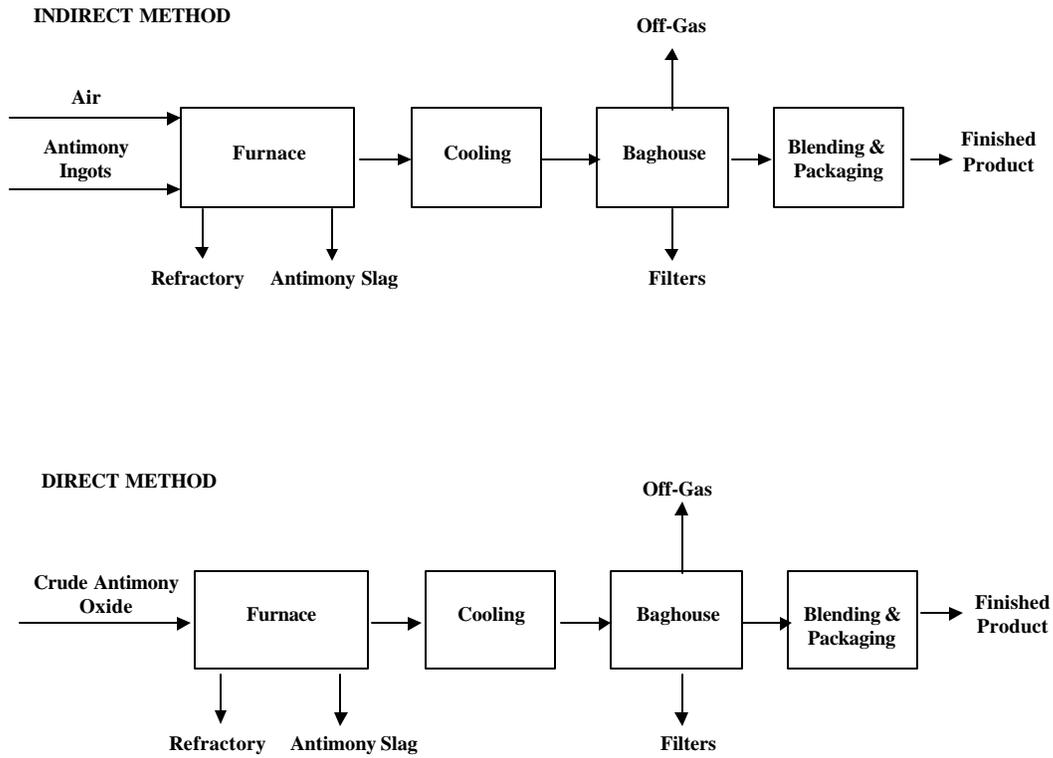
Antimony Oxide Production via the Direct Method

In this process, crude (low grade) antimony oxide is roasted in the presence of air to produce antimony oxide vapors. The vapors are condensed to generate antimony oxide powder. A baghouse is used to recover product in the off-gas. The antimony oxide powder is then blended, classified and bagged for shipment. The crude antimony oxide comes either from off-site or is reclaimed from secondary materials from within the facility.

2.2 PRODUCTION TRENDS, CHANGES AND IMPROVEMENTS

In the response to the RCRA § 3007 Survey, U.S. Antimony reported plans to use a different type of refractory, which may eliminate or reduce the generation of refractory at the facility. Great Lakes Chemical Corporation also reported plans to start sending the empty supersacks generated at their facility to a plastics recycling facility.

Figure 2.1 Process Flow Diagram for the Production of Antimony Oxide



3. RESIDUAL GENERATION AND MANAGEMENT

This section discusses the wastes and materials generated from the production of antimony oxide. For the purposes of this listing determination, the residuals generated as a result of the production of antimony oxide were grouped into categories. **Section 3.1** presents a detailed discussion of the production steps that generate the wastes, the management steps for the wastes, a characterization of the physical and chemical properties of the wastes, and results of initial screening analysis. **Section 3.2** describes the residuals categories that are outside the scope of the consent decree. **Section 3.3** describes materials that are reused in the production of antimony oxide, either on-site or off-site. **Appendix A** presents a complete summary of the residuals generated at each of the facilities and their management.

3.1 EVALUATION OF ANTIMONY OXIDE WASTE CATEGORIES

3.1.1 Antimony Slag Not Reclaimed in Antimony Oxide Process

Waste Generation and Management

All four facilities reported generating a slag in their furnaces due to the impurities in the feedstock. Generally, the slag is stored in containers or containment buildings prior to being reinserted into the furnace (either on-site or off-site) for continued use in antimony oxide production.

However, some slag is too low in antimony to be reused in the antimony oxide process. Laurel Industries, U.S. Antimony, and Amspec Chemical Corporation reported generating a slag that is not reused in the antimony oxide process. Although U.S. Antimony reported that they intend to recycle these low antimony slags, they have stored this slag in drums at the facility for the last four to 10 years. The facility described plans to place the slag in an on-site disposal unit upon construction completion. Amspec Chemical Corporation stores their slag in containers prior to selling it to lead smelters for antimony recovery. Laurel Industries stores slag in containers before sending it off-site for lead recovery. **Table 3.1** presents a summary of the final management step for the antimony slag not reused in the antimony oxide process at all three facilities. The numbers reported in the second column represent the total number of wastes that were reported with the final management step identified. **Appendix A** contains the total of volume slag generated (including recycled volumes) for each of the four facilities.

Table 3.1 Waste Management Summary for Antimony Slag Not Recycled in the Antimony Oxide Process

Final Management	# of Wastestreams with Reported Volumes	Total Volume (MT/yr)
Long term on-site storage in containers	1	20
Sent for metals recovery (e.g. lead or non-antimony oxide production)	2	93

Waste Characterization

The constituents of concern reported in the RCRA §3007 survey for the slag included antimony

arsenic, and lead at various concentrations. Silica was also a reported constituent. Laurel Industries reported a D008 (lead) waste code for their slag.

Three samples were collected during record sampling to characterize the antimony slag waste category:

Sample	Facility	Description
AC-1-AO-01	U.S. Antimony	Collected from drums of "Reduction furnace slag" that were designated as containing less than 5 percent antimony.
AC-1-AO-06	U.S. Antimony	Collected from drums of "Reduction furnace slag" that were designated as containing 5 to 10 percent antimony.
LI-1-AO-01	Laurel Industries	Collected from drums used to collect the material.

The following related documents have also been included in the docket:

- S *Laurel Industries, Inc., Site Sampling and Analysis Plan for Record Sampling under the Inorganic Listing Determination* - September 13, 1999
- S *United States Antimony Corporation, Site Sampling and Analysis Plan for Record Sampling under the Inorganic Listing Determination* - September 22, 1999
- S *Waste Characterization Report, Laurel Industries Inc., LaPorte, Texas* - February 1, 2000
- S *Waste Characterization Report, U.S. Antimony Corporation, Thompson Falls, Montana* - November 29, 1999

Totals, TCLP and SPLP analyses were conducted on the samples. **Table 3.2** lists the analytical results for the constituents detected in the samples. The validated analytical data reports for these samples can be found in the two Waste Characterization Reports.

Table 3.2 Characterization of Antimony Slag from the Production of Antimony Oxide

Parameter	AC-1-AO-01			AC-1-AO-06			LI-1-AO-01			SSL mg/kg	HBL mg/l
	Total mg/kg	TCLP mg/l	SPLP mg/l	Total mg/kg	TCLP mg/l	SPLP mg/l	Total mg/kg	TCLP mg/l	SPLP mg/l		
Antimony	11500 ^l	55.8	114	12700 0 ^l	110	211	84700	24.6	21.8		0.006 3
Arsenic	301	2.0	2.93	478	3.1	3.81	69.2 ^l	<0.5 ^u	0.0239	4.7	0.000 74
Barium	294	<2	<2	257	<2	<0.1	61.8	<2 ^u	<2 ^u	5,600	1.1
Beryllium	<20	<0.02	0.0034 ^l	<100	<0.02	0.0024 ^l	0.5	<0.02 ^u	<0.002 ^u	160	0.031
Boron	<500	9.8 ^k	9.27	<2500	8.5 ^k	8.06 ^l	158 ^k	<2 ^{u,l}	0.100	7,200	1.4
Cadmium	<50 ^u	<0.05 ^u	<0.005 ^u	<250 ^u	<0.05 ^u	<0.005 ^u	12.8 ^k	<0.05 ^u	<0.005 ^u	40	0.007 8
Calcium	8130	20.1	3.42	9000	17.0	4.46	412	2.8	2.00	N/A	
Chromium	<50 ^u	<0.05 ^u	<0.005 ^u	<250 ^u	<0.05 ^u	<0.005 ^u	28.3 ^k	<0.05 ^u	<0.005 ^u	120,00 0	23
Chromium 6 ⁺	<0.02 ^u	<0.50 ^u	<0.50 ^u	<0.02 ^u	<0.20 ^u	<0.50 ^u	<0.02 ^u	0.04	<0.02 ^u	240	0.047
Cobalt	<50	<0.05	0.0061	<250	<0.05	<0.005	55.0 ^l	<0.05 ^u	<0.005 ^u	4,800	0.94
Copper	52.2 ^k	<0.25	0.0087	<250	<0.25	0.0079	4610 ^l	6.3 ^l	0.294	19	1.3
Iron	13600	1.3	0.662 ^l	13500	8.8	2.87 ^l	9780	1.5	0.179	430,00 0	5
Lead	135 ^l	<0.5	<0.005	491 ^l	<0.5	<0.005	34000	22.3	1.58	400	0.015
Manganese	160 ^k	<0.05	<0.005	<250	<0.05	<0.005	449	0.1	0.0242	3,800	0.73
Mercury	<0.1	<0.00 2	<0.0002	<0.1	<0.00 2	0.0003	<0.1 ^u	<0.002 ^u	<0.0002 ^u	24	0.004 7
Nickel	<50 ^u	<0.2 ^u	<0.005 ^u	<250 ^u	<0.2 ^u	<0.005 ^u	488	0.5	0.0241	1600	0.31
Potassium	2020	89.6	87.6	1980	83.6	83.4	1560	<10 ^u	<1	N/A	
Selenium	<50	0.6	0.550	<250	0.6	0.331	<0.5 ^k	<0.5 ^u	<0.005 ^u	400	0.078
Silver	<10 ^u	<0.1 ^u	<0.001 ^u	<100 ^u	<0.1 ^u	<0.001 ^u	465 ^l	<0.1 ^u	0.0226 ^l	400	0.078
Sodium	32100 0	N/A	13000	26200 0	N/A	11100	4160 ^k	N/A	10.0	N/A	
Thallium	<200 ^u	<2 ^u	<0.005 ^u	<1000 ^u	<2 ^u	<0.005 ^u	3.8	<2 ^u	<0.005 ^u	6.4	0.001 3
Titanium	2440	0.067	<0.005 ^l	761	0.2	<0.005 ^l	83.6	<0.05 ^u	<0.005 ^u	N/A	
Vanadium	<50	1.3	1.14	<250	0.6	1.0	9.6	<0.05 ^u	<0.005 ^u	720	0.14
Zinc	<500 ^u	<3 ^u	<0.05 ^u	<2500 ^u	<3 ^u	<0.100 ^u	1360	<2 ^u	0.0533	24,000	4.7
Specific gravity	2.7	N/A	N/A	2.6	N/A	N/A	4.8	N/A	N/A		
Moisture content, %	<2	N/A	N/A	<2	N/A	N/A	<2 ^u	N/A	N/A		
Final pH of leachate	N/A	11.6	12.9	N/A	10.8	12.7	N/A	4.99	7.51		

N/A: not analyzed

J: analyte present - RPD is outside QC limit

K: analyte present - reported value may be biased high

L: analyte present - reported value may be biased low

Results of Initial Screening Analysis and Assessment of Management Scenarios

Our risk evaluation focused on the antimony slag that is accumulated and held for several years. U. S. Antimony has been placing approximately 20 MT/yr in steel drums on pallets on the ground for a number of years. This waste has been stored on-site with the facility reporting that they intend to reclaim the antimony from this slag when antimony prices are favorable. The facility's Operating Permit⁸ requires that the slag be placed in an on-site engineered "slag storage pit" in the future. Based on this information, an on-site landfill scenario for this waste was assessed.

The slag that is reinserted into the antimony oxide process either on-site or off-site is stored in containment buildings or containers for limited periods of time prior to being reused in the process. The slag that is sent to lead facilities is also stored in containers for short periods of time prior to being sent off-site for recycling. We did not evaluate these scenarios further because these materials are managed prior to reuse in ways that present a low potential for release, and because we evaluated the in-scope wastes generated after reuse of these materials. For the slags that go to lead facilities, we believe that the lead wastes generated by these facilities are beyond the scope of the consent decree.

In evaluating the slag that is accumulated for several years, we used the total and SPLP results for samples AC-1-AO-01 and AC-1-AO-06, reflecting the industrial nature of U.S. Antimony's projected management practice for this waste in the on-site unit. The analytical data for the sample collected at Laurel Industries was not used because the sample is TC hazardous for lead and could not, therefore, be sent to a Subtitle D landfill. Also, Laurel's waste is sent for recycling. Both of the U. S. Antimony samples are relevant because they represent the material stored on-site and the material destined for the on-site slag pit.

The following constituents exceeded HBLs in the SPLP, as illustrated in **Table 3.2**:

- antimony
- arsenic
- boron
- selenium
- vanadium

Based on these findings, the EPA determined that further modeling was required. The EPA conducted modeling of the onsite industrial landfill scenario. Only the annual volume reported to be stored on-site indefinitely (20 MT) was used for the groundwater modeling of this waste. The facility reported that it processes sodium antimonate from two facilities and returns the resultant slag to the process for further processing. These recycled slag volumes were not considered.

⁸ *"United States Antimony Corp. Stibnite Hill Mine Project Operating Permit 00045", 6th review draft, January 1999.*

Information available on the Internet for the State of Montana⁹ (where the slag is stored) indicates that groundwater is a viable and actively used resource in this area. See **Appendix B** (hard copy only) for the well details from the web page and the TopoZone map showing the placement of the wells. Because the groundwater is used for domestic purposes, we assessed potential releases to residential wells. Refer to the “Risk Assessment for the Listing Determinations for Inorganic Chemical Manufacturing Wastes” (August 2000) for more details and the results of this analysis.

3.1.2 Baghouse Filters

Waste Generation and Management

Antimony oxide vapors are filtered through baghouse filters during the production process. These filters capture product or off-specification product. The filters from the baghouse are changed periodically at all four facilities. U.S. Antimony generates filters in both their reduction and oxidation furnaces.

All four facilities reported different management practices for this waste. Laurel Industries sends this waste to an off-site Subtitle D industrial landfill. Amspec Chemical Corporation sends this waste to an off-site non-hazardous waste incinerator. U.S. Antimony and Great Lakes recycle their filters into their on-site production process for antimony recovery. U.S. Antimony also sends a portion of their filters to Mexico for antimony recovery. **Table 3.3** provides a summary of the final management of the baghouse filters.

Table 3.3 Waste Management Summary for Baghouse Filters

Final Management Step	# of Wastestreams Reported	Total Volume (MT/yr)
Off-site Industrial Subtitle D landfill	1	4
Off-site non-hazardous waste incinerator	1	3
Sent to Mexico for antimony recovery	2	2
Sent to on-site furnace for antimony recovery	1	NR

Waste Characterization

Three samples were collected during record sampling to characterize this waste. At U.S. Antimony,

⁹ <http://www.deq.state.mt.us>. Sanders County

one sample (AC-1-AO-03) was collected from a drum containing the “oxidation furnace” baghouse filters and one sample (AC-1-AO-07) was collected from a drum containing the “reduction furnace” baghouse filters. At Laurel Industries, the sample (LI-1-AO-03) was of the baghouse filters associated with the facility’s kiln. The samples collected represent the range of production practices in the industry. At both facilities, the samples were collected by cutting sections of baghouse filters stored in drums. The following related documents have also been included in the docket:

- S *Laurel Industries, Inc., Site Sampling and Analysis Plan for Record Sampling under the Inorganic Listing Determination - September 13, 1999*
- S *United States Antimony Corporation, Site Sampling and Analysis Plan for Record Sampling under the Inorganic Listing Determination - September 22, 1999*
- S *Waste Characterization Report, Laurel Industries Inc., LaPorte, Texas - February 1, 2000*
- S *Waste Characterization Report, U.S. Antimony Corporation, Thompson Falls, Montana - November 29, 1999*

Totals, TCLP and SPLP analyses were conducted on the samples. **Table 3.4** lists the constituents that were detected in the three samples. The validated analytical data reports for these samples can be found in the two Waste Characterization Reports.

Table 3.4 Characterization of Baghouse Filters from Antimony Oxide Production

Parameter	LI-1-AO-03		AC-1-AO-03		AC-1-AO-06		HBL (mg/l)	TC mg/l
	TCLP (mg/l)	SPLP (mg/l)	TCLP (mg/l)	SPLP (mg/l)	TCLP (mg/l)	SPLP (mg/l)		
Antimony	9.3	6.2	9.9	4.3	68.7	287	0.0063	
Arsenic	<0.5	0.6	<0.5	0.09	6.9	6.9	0.00074	5.0
Boron	6.5	1.0	<2	0.2	<2	0.7	1.4	
Cadmium	0.3	0.5	0.3	0.3	<0.05	0.9	0.0078	1.0
Lead	8.5	16.9	2.8	1.0	<0.5	<0.05	0.015	5.0
Mercury	<0.002	0.001	<0.002	<0.0002	0.03	0.4	0.0047	0.2
Thallium	<2	0.06	<2	0.06	<2	0.1	0.0013	

Assessment of Management Scenarios

Per the data presented above in **Table 3.4**, the analysis of samples LI-1-AO-03 and AC-1-AO-03 indicates that this waste fails the TC for lead and arsenic. Other metals with minor exceedences of the HBLs include hexavalent chromium, copper, selenium and silver. None of the identified their wastes as exhibiting the toxicity characteristic. Laurel and Amspec both manage this waste as non-hazardous. U.S. Antimony and Great Lakes recycle the filters either by returning them to the on-site

production process or sending them off-site for reclamation.

3.1.3 Empty Supersacks

Great Lakes Chemical Corporation was the only facility that reported generating this waste. The empty supersacks are generated when the bags used for antimony ore are no longer used to hold the raw material. The facility reported generating 15 MT/yr and sending it off site to a subtitle D landfill or a plastic recycler. No samples of this waste were collected.

The facility claims that the supersacks are empty and would meet the standard in 40 CFR 261.7 (which exempts “empty” containers formerly used to manage hazardous waste). These supersacks were not evaluated further.

3.1.4 Floor Sweepings, Spills, Off-spec Product and Other Residuals

All four facilities reported generating and collecting other material containing antimony at various points of the production process including floor sweepings, spills, classifier bottoms, ductwork cleanouts, off-specification product and truck wash sludge and reinserting this material into the antimony oxide process. None of these materials were reported as hazardous. Three of the four facilities reported recycling these materials back into the process on-site. Great Lakes Chemical Corporation reported storing these materials in containers prior to sending them off-site for use producing antimony oxide. The reported volumes and a summary of the management for these materials is presented in **Appendix A**. These materials are reused in production units and there are no significant pathways for exposure of these materials to the environment prior to their reuse. These materials were stored in containers or in containment buildings. Therefore, we did not evaluate them further for purposes of this listing.

3.2 DEBRIS AND OTHER NON-PROCESS WASTES

The EPA does not consider some kinds of debris and plant component materials to fall within the scope of the consent decree. Most of the wastes that fell into this category were refractory brick wastes generated when facilities refurbish plant furnaces. All four of the antimony oxide producers reported generating furnace refractory. EPA considers this material to be a structural component of the plant where production takes place rather than a waste from the “production” of antimony oxide.

APPENDIX A

Summary of Waste Generation and Management

and

Summary of Materials Recycled into the Antimony Oxide Production Process

Table A.1 - Wastes from the Production of Antimony Oxide

Waste Category	Facility	Waste Volume (MT/yr)	Final Waste Management Step
Antimony slag not recycled in antimony oxide process	U.S. Antimony	20	Stored in containers
	Amspec	20	Sold to Mexican broker for lead/antimony recovery
	Laurel Industries	73	Sent off site for lead recovery
Baghouse filters	Laurel Industries	4	Sent to Subtitle D landfill
	Amspec	3	Sent to Off-site non-hazardous waste incinerator
	U.S. Antimony	2	Sent to on-site Sb recovery furnace or Sent to Mexico for Sb recovery
	Great Lakes	NR	Sent to on-site Sb recovery furnace
Empty supersacks	Great Lakes	15	Sent to Subtitle D landfill or plastic recycler

Table A.2 Materials Recycled in the Antimony Oxide Process

Facility	Total Volume (MT/yr)	Management
Great Lakes Chemical Corporation		
Furnace tappings and drippings (slag)	650	Sent to Mexico for antimony recovery. Recovered antimony sent back to GLCC for reuse in antimony oxide production process.
Floor sweepings and product spills	3	
Truck wash sludge	3	
Oxide dust collected in hygiene system	12	Recycled back into process
Coarse and off-specification product	250	Recycled back into process
Laurel Industries Inc.		
High antimony slag	544	Recycled back into process
Floor sweepings and product spills	27	
Amspec Chemical Corp		

Facility	Total Volume (MT/yr)	Management
Sawtooth (high antimony slag)	10	Recycled back to process
Residual product from furnace	3	
Classifier bottoms	50	
Off-specification product	50	
U.S. Antimony		
High antimony slag	2	Recycled back to process
Residual product from cooling tower	50	
Residual product from baghouses	10	
Residual product from wash stations	0.25	
Floor sweepings	10	Recycled back to process

APPENDIX B
Groundwater Well Information for U.S. Antimony